Characterization of Activated Carbon and its Performance to Treat Water Used for Enhanced Oil Recovery

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Abstract: Oil recovery operations traditionally have been subdivided into three stages: primary, secondary and tertiary (Enhanced Oil Recovery EOR). Primary production, the initial production stage, resulted from the displacement energy naturally existing in a reservoir. Traditionally secondary recovery processes are water flooding, pressure maintenance, and gas injection. Tertiary recovery (EOR) processes involve the injection of a fluid of some type into a reservoir. The injected fluids and injection processes supplement the natural energy present in the reservoir to displace oil to a producing well. In addition, the injected fluids interact with the reservoir rock/oil system to create conditions favorable for oil recovery. The interactions are attributable to physical and chemical mechanisms and to the injection or production of thermal energy. In the present research work a different sizes of activated carbon extracted from date nuclei were used spontaneous imbibition test for sandstone core samples after saturated crude oil. A spontaneous imbibition test consisting of three scenarios of activated carbon at various temperatures and later compared it with one scenario of untreated reservoir water. In this study the spontaneous imbibition test was performed at room temperature and oven temperature including: (45, 55, and 70℃). The results showed that the best oil recovery was at a treated water with activated carbon volume (0.125), which was (70.41%), that was at the maximum used temperature.

Keywords: Activated Carbon; Reservoir Water; treatment process;; Spontaneous imbibition test.

وصف الكربون المنشط وأدائه في معالجة المياه المستخدمة في تحسين إنتاج النفط

محمد صمبة, مسعودة فرحات, محمود الشريف, محمد المدني, الفيتوري خليفة, , طه عبدالله, طه أبراهيم, محمد ميلاد, حاتم الدرازي

الخلاصة: بصورة تقليدية تم تقسيم عمليات استخلاص النفط الى ثلاث مراحل: أولية وثانوية وثلاثية (تحسين استخلاص النفط). تعتمد مرحلة الإنتاج الأولية على الطاقة الموجودة بصورة طبيعية في المكمن. وتعتمد عملية الإنتاج الثانوية على الغمر بالمياه, والمحافظة على الضغط, والحقن بالغاز. وتتضمن عمليات استخلاص النفط الثلاثية على حقن سائل من نوع معين في الخزان, حيث ان هذه السوائل تكمل الطاقة الطبيعية الموجودة في الخزان وبالتالي تؤدي الى تحسين انتاج النفط, وبصورة أخرى تتفاعل السوائل التي تم حقنها في الخزان مع صخور وزيوت المكن وتعمل على استعادة النفط. ويعزى حدوث هذه التفاعلات الى الخواص الفيزيائية والكيميائية التي تؤدي الى انتاج طاقة حرارية. في هذه الدراسة البحثية تم استخدام احجام مختلفة من الكربون المنشط, والذي تم استخلاصه من نواة التمر, وذلك لاختبار التشرب التلقائي لعينات من الحجر الرملي يعد تشبعها بالنفط الخام. اختبار التشرب التلقائي يتكون من ثلاث سيناريوهات للكربون المنشط عند درجات حرارة مختلفة ومقارنتها لاحقاً بسيناريو واحد لمياه الخزان الغير معالجة. في هذه الدراسة, تم اجراء اختبار التشرب التلقائي عند درجة الحرارة العادية وكذلك عند درجات الحرارة 45 و 55 و 70 درجة مئوية. من خلال النتائج المتحصل عليها تبين أن أفضل استخلاص للنفط كان في المياه المعالجة بالكربون المنشط والذي بحجم 0.125 والذي كانت نسبته 70.41% وكان ذلك عند أقصى درجة حرارة مستخدمة.

1. Introduction

After more than a century of petroleum exploitation, thousands of oil and gas fields are approaching the ends of their economically productive lives. The life of an oil well usually undergoes three distinct phases where

various techniques are employed to maintain crude oil production at maximum levels which are primary, secondary and tertiary [1]. These techniques are mainly aimed to serve a common purpose which is reducing the residual oil saturation which results in enhancement of the displacement efficiency and improvement of the ultimate oil recovery. Primary oil recovery refers to the process of extracting oil either via the natural rise of hydrocarbons to the surface of the earth or via pump jacks and other artificial lift devices. Since this technique only targets the oil, which is either susceptible to its release or accessible to the pump jack. This is very limited in its extraction potential. In fact, only around 5% - 15% of the well's potential are recovered from the primary method.

In the primary stage, the pressure of the reservoir plays a vital role because it causes the proper flow of oil towards the surface. This pressure depends on the internal energy and properties of the hydrocarbon fluids [1]. This natural force includes the driving energy of water from a large aquifer (a natural underground water flow), i.e., aquifer drive, dissolved gas drive, gas cap drive, and expansion by rock and fluid. Because this technique solely targets the oil and its susceptibility or accessibility, it has limited extraction potential [2,3].

Secondary recovery stage involves the injection of water or gas into the reservoir to restore the pressure or to maintain the movement of oil in the reservoir. This is undertaken when the pressure decreases due to the gradual production of oil in the reservoir, and the low pressure is no longer able to push the oil to the surface. At this stage, a critical point is reached when external energy is needed for the recovery of oil. Gas injection is mainly used with a gas cap to displace the oil in reservoirs. The pores of the rocks are prevented from clogging via the use of dissolved gases in the water used for flooding. At this point, the viscosity of the oil is minimized, consistent with thermodynamics. In this situation, a maximum quantity of oil is displaced under a specifically maintained gradient of pressure. At subsequent stages, when the quantity of oil relative to the water increases, it is no longer economical to continue the process, and the oil reserve should be abandoned [3,4,5]. Around 50% to 70% of the world's unrecoverable crude oil remains in reserves that have been

exhausted by conventional methods [6–8].

The only objective of this stage is to maximize the utilization of the crude oil that cannot be recovered during the previous oil recovery stages. This includes both residual oil and the remaining oil. The former refers to the immobile crude oil residue in the reservoir pores, whereas the latter refers to un-swept crude oil (which is stuck due to the formation of heterogeneity). This step is used in fields that are characterized by poor permeability, heavy oil, or irregular fault lines; it either alters the reservoir fluid properties or the rock's characteristics, to make the reservoir more conducive to extraction. The economics of this method must be valid. Therefore, to determine which type of EOR will suit the reservoir, each field must be precisely evaluated by screening, reservoir characterization, scoping, and reservoir modelling, in addition to simulation [9]. This stage aids in improving the crude oil's mobility, the properties of the oil-water interface, and the microscopic and macroscopic sweep efficiency. Further extensive literature and laboratory research is required on the fluid, the reservoir rocks, and the individual processes of this stage [9]. In this stage, oil can be recovered using several thermal methods, such as steam flooding [10] and in situ combustion or fire flooding
[11]; chemical methods using alkaline displacement, surfactants, and polymers [12]; miscible displacement methods using CO_2, N_2, flue gases, kerosene, gasoline, benzene, etc. [13]; and via the microbial method.

Activated carbons are made from materials rich in carbon through carbonization and an activation process. A porous structure and its adsorption properties can be obtained in carbonaceous materials via either chemical or physical1 activation. Physical activation involves pyrolysis of source material at 800 ºC – 1000 ºC to produce charcoal. This is then followed by activation using steam, carbon dioxide or oxygen [14].

Chemical activation involves the impregnation of the precursor material with a chemical activating agent followed by activation at temperatures of 400 ºC – 700 ºC under nitrogen atmosphere2. Zinc chloride ($ZnCl_2$), potassium hydroxide (KOH) and phosphoric acid (H_3PO_4) are widely used as chemical activating agents. The activating chemical agents influence the

pyrolytic decomposition and inhibiting formation of tar and volatile matter, thus enhancing the yield of Activated carbon [15]. The dehydration and oxidation characteristics of chemical activation agent require much lower activation temperature compare to physical activation [16]. The aim of this research is to produce activated carbon from the local agricultural waste which is local palm date impregnated with two activation agents for a particular application. To achieve these, a study was carried out with the following objectives: (a) To get the best oil recovery from rock; (b) To evaluate various operating parameters such as activation temperature and activation time for the activated carbon produced from local palm date; (c) Knowing the effectiveness of activated carbon and their effect on increasing the extraction of oil from sand rocks (d) To evaluate the potential application of locally produced activated carbon in produced water for injection water. The overall project plan of the present work is shown in figure (1.1).

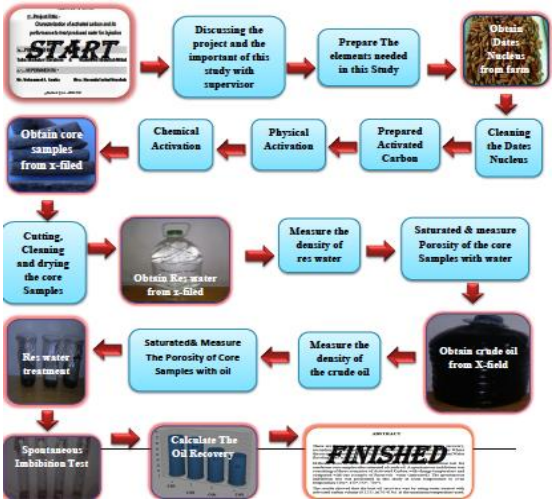
START
Discussing the project and the important of this study with supervisor
Prepare The elements needed in this Study
Obtain Dates Nucleus from farm
Cleaning the Dates Nucleus
Prepared Activated Carbon
Physical Activation
Chemical Activation
Obtain core samples from x-filed
Cutting, Cleaning and drying the core Samples
Obtain Res water from x-filed
Measure the density of res water
Saturated & measure Porosity of the core Samples with water
Obtain crude oil from X-field
Measure the density of the crude oil
Saturated& Measure The Porosity of Core Samples with oil
Res water treatment
Spontaneous Imbibition Test
Calculate The Oil Recovery
FINISHED

Figure (1.1): The Overall Project Plan

2. Experiments Procedure

2.1 Activated Carbon Preparation

2.1.1. First Stage of Activated Carbon Preparation

(i) Date nucleus were washed several times using distilled water until the color of the water becomes clear as shown in figure (2.1.a), (ii) Dates nucleus were placed in burning oven at 400°C for a period of 24 hours. The nucleus were completely burned and their color were changed to dark black as shown in figure (2.1.b), (iii) After that, the Dates Nucleus were grounded into fine powered parts as shown in figure (2.1.c). (iv) The grounded nucleus were washed several times using distilled water as shown in figure (2.1.d), (v) The grounded particles were put in drying oven at 105°C for a period of 24h as shown in figure (2.1.e), (vi) After that, the grounded powders were classified into three different size

(0.250, 0.125, 0.063) mm, and so the resulted samples as shown in figure (2.1.f) were a non-activated carbon.

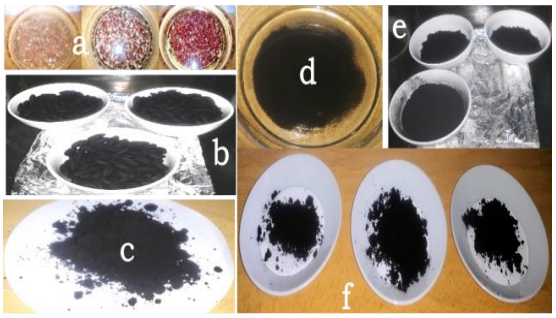

Figure (2.1) Carbon Activation First Stage

2.1.2 Second Stage of Activated Carbon Preparation

The non-activated carbon is activated by using a chemicals such as zinc chloride ($ZnCl_2$), sulfuric acid (H_2SO_4), and also sodium hydroxide (Na OH). The used chemical for activation are shown in figure (2.2).The second stage of activated carbon preparation was carried out as the following: (i) A (10 g) of non-activated charcoal sample was add to a mixture of (H_2SO_4 (3N) and the $ZnCl_2$ (3N) in 50 ml of distilled water) as shown in figure (2.2.a), (ii) A non-activated carbon mixture was exposed to boiling temperature (100C°) for a period time of 3 hours as shown in figure (2.2.b) thus, the activated carbon was formed, (iii) The formed activated carbon was filtered as shown in figure (2.2.c). (iv). The collected sample of activated carbon was washed using a distilled water for a several times and after each washing process a pH was measured using a pH measurement device as shown in figure (2.2.d). The required pH value was 7 . (v) In order to dried the activated carbon, the samples were inserted in drying oven at temperature of (105C°) for a period time of 24 hours as shown in figure (2.2.e). The activated carbon samples were ready for a reservoir water treatment as shown in figure (2.2.f).

3. Experimental Results and Discussion

3.1 Reservoir Water and Oil Analysis

3.1.1 Reservoir Water Analysis Before the Treatment Process

Table (3.1) shows the results of reservoir water analysis before the treatment process.

Table (3.1) Reservoir Water Analysis Before the Treatment Process

S.No	Test Type	Result
1	Ec	2584
2	Salinity	24 ppt
3	PH	6.77
4	TDs	22830 mg/l
5	NH_4	0.15 mg/l or ppm
6	NO_3	2.820 ppm
7	Fe	< 0.001 ppm
8	K	600 ppm
9	Ca	6535 ppm
10	Mg	4130 ppm
11	SO_4	2720 ppm
12	Cl	138.2 ppm
13	Na	11920 ppm

3.1.2 Reservoir Water Analysis After the Treatment Process

Table (3.2) shows the results of reservoir water analysis after the treatment process.

3.2 Density Results

3.2.1 Water Density Results

Table (3.3) exhibits the results of density for the natural reservoir water and treated reservoir water.

3.2 Density Results

3.2.1 Water Density Result

Table (3.3) presents the results of density for the natural reservoir water and treated reservoir water.

Table (3.2) Reservoir Water Analysis After the Treatment Process

S. No.	Test Type	Result		
		0.250 mm	0.125 mm	0.063 mm
1	PH	6.90	6.96	6.88
2	Ec	50.90 ms/cm	48.76 ms/cm	59.24 ms/cm
3	Salinity	22 ppt	20 ppt	22 ppt
4	TDs	2007 ppm	19483 ppm	19870 ppm
5	NH_4	0.11 ppm	0.16 ppm	0.13 ppm
6	NO_3	0.98 ppm	0.02 ppm	0.14 ppm
7	Fe	<0.001 ppm	<0.001 ppm	<0.001 ppm
8	K	10 ppm	8.40 ppm	9.5 ppm
9	Ca	7 ppm	9 ppm	6 ppm
10	Mg	57 ppm	10 ppm	10 ppm
11	SO_4	197 ppm	158 ppm	171 ppm
12	Cl	12 ppm	24 ppm	27 ppm
13	Na	13 ppm	41 ppm	62 ppm

Table (3.3) Density for the Natural and Treated Reservoir Water

Water Type		W_{dry} (g)	W_{sat} (g)	V_w (cc)	ρ_o (g/cc)
Natural Reservoir Water		67.797	138.735	70	1.0134
Treated Reservoir Water	0.250mm	67.797	138.357	70	1.0080
	0.125mm	67.797	138.000	70	1.0029
	0.063mm	67.797	138.21	70	1.0059

Table (3.4) showing the results of density for the reservoir oil.

Table (3.4) Density for the Reservoir Oil

Oil Type	W_{dry} (g)	W_{sat} (g)	V_o (cc)	Density (g/cc)
X-field	71.7	138.5	70	0.797

3.3 Core Analysis Results

3.3.1 Pore Volume of Sandstone Core Samples Results

The volume of fluids type which inter to pore volume of sandstone reservoir core samples space were calculated. Table (3.5) showing the results of pore volume with reservoir water. Table (3.6) showing the results of pore volume with reservoir oil.

Table (3.5) Pore Volume of Sandstone Core with Reservoir Water

Sample	W_{dry} (g)	W_{sat} (g)	ρ_w (g/cc)	PV (cc)
C-101	62.8	70.4	1.0134	07.49
C-102	59.9	69.3		09.27
C-103	53.6	64.3		10.55
C-104	63.2	71.7		08.38
C-105	63.7	71.7		07.89
C-106	63.6	71.8		08.09

Table (3.6) Pore Volume of Sandstone Core with Reservoir Oil

Sample	W_{dry} (g)	W_{sat} (g)	ρ_o (g/cc)	PV (cc)
C-101	62.8	69.3	0.797	8.155
C-102	59.9	67.7		9.786
C-103	53.6	62.6		11.29
C-104	63.2	70.6		9.284
C-105	63.7	70.6		8.657
C-106	63.6	70.8		9.033

3.3.2 Bulk Volume of Sandstone Core Sample Results

The bulk volume of carbonate and sandstone reservoir core samples at different volumes were calculated using the cylinder volume base. Table (3.7) shows the bulk volume of sandstone core samples.

Table (3.7) Bulk Volume of Sandstone Core Samples

Sample	L (cm)	D (cm)	V_b (cc)
C-101	4.919	2.906	55.22
C-102	4.921	2.050	55.25
C-103	4.916	2.835	53.81
C-104	4.915	2.915	55.30
C-105	4.910	2.905	55.00
C-106	4.915	2.915	55.30

3.3.3 Porosity of Sandstone Core Sample Results with Reservoir Water and Oil

The porosity was obtained by dividing the pore volume by the bulk volume. Table (3.8) and (3.9) show the porosity of sandstone core samples for reservoir water and oil.

Table (3.8) Porosity of Sandstone Core Samples of reservoir Water

Sample	V_b (cc)	V_p (cc)	Ø	
			%	Fra
C-101	55.22	07.49	13.5	0.135
C-102	55.25	09.27	16.7	0.167
C-103	53.81	10.55	19.6	0.196
C-104	55.30	08.38	15.1	0.151
C-105	55.00	07.89	14.3	0.143
C-106	55.30	08.09	14.6	0.146

According to the obtained results, the maximum porosity of carbonate core sample in reservoir water was 19.6% at sample C-103, while the maximum porosity of sandstone cores samples was 13.5% at sample C-101. Moreover and based on the obtained results, the maximum porosity of carbonate core sample was 20.2% at sample C-103, while the maximum porosity of sandstone cores samples was 14.7% at sample C-101.

Table (3.9) Porosity of Sandstone Core Samples of Reservoir Oil

Sample	V_b (cc)	V_p (cc)	Ø	
			%	Fra
C-101	55.22	8.155	14.7	0.147
C-102	55.25	9.786	17.7	0.177
C-103	53.81	11.29	20.2	0.202
C-104	55.30	9.284	15.7	0.157
C-105	55.00	8.657	15.7	0.157
C-106	55.30	9.033	16.3	0.163

3.3.4 Original Oil In Place (OOIP) in Sandstone Core Results

Table (3.10) shows original oil in place (OOIP) of sandstone core samples, some core samples were saturated with oil with density of 0.797 g/cc. Based on the obtained results, the maximum PPIO of carbonate core sample was

11.29cc at sample C-103, while the maximum PPIO of sandstone cores samples was 8.155 cc at sample C-101.

3.4. Spontaneous Imbibition Test

3.4.1 Effect of Natural Reservoir Water, Time, and Temperature on the Oil Recovery of Core Samples

The spontaneous imbibition test was done by the reservoir water (untreated water), and the used sample was (C-001). At the beginning the temperature was about (27˚C) for a period time of 24 hours, thus, the oil extraction rate (OER) was (0.24%), by increasing the temperature to (45˚C) for a period of 72 hours, (OER) was increased to (8.82 %), farther the test was carried out for another 72 hours at the same temperature. In this case (OER) was (22.31 %). Finally. the temperature was raised to (70 ˚C) for 72 hours and (OER) was (46.8 %). Figure (3.1) show the effects of reservoir water (untreated) on the oil recovery for sandstone core samples as function of time and temperature.

Table (3.10) (OOIP) in Sandstone Core Results

Sample	W_{dry} (g)	W_{sat} (g)	ρ_o (g/cc)	OOIP (cc)
C-101	62.8	69.3	0.797	8.155
C-102	59.9	67.7		9.786
C-103	53.6	62.6		11.29
C-104	63.2	70.6		9.284
C-105	63.7	70.6		8.657
C-106	63.6	70.8		9.033

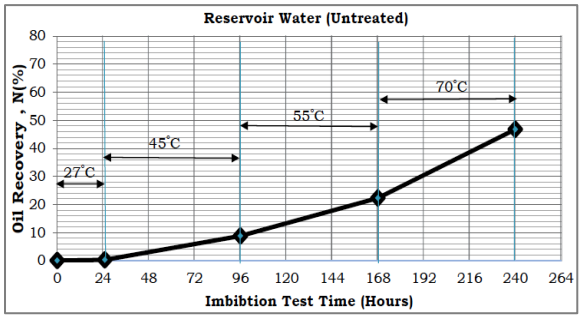

Figure (3.1) Effects of Reservoir Water (Untreated) on Oil Recovery of Sandstone Core Samples Related with Time and Temperature

3.4.2 Effect of Treated Reservoir Water, Time, and Temperature on the Oil Recovery of Core Samples

(i) First Scenario; Reservoir Water was Treated by Activated Carbon (0.250 mm)

The spontaneous imbibition test was done by the reservoir water treated by activated carbon with size of 0.250 mm, and the used sample was (C-002). At the beginning the temperature was about (27 ˚C) for a period time of 24 hours, thus, the oil extraction rate (OER) was (0.40 %), by increasing the temperature to (45 ˚C) for a period of 72 hours, (OER) was increased to (15.73 %). Then the test was carried out for

addition 72 hours at the same last temperature. In this case (OER) was (34.13 %). Finally. the temperature was raised to (70˚C) for 72 hours and (OER) was (68.87%). Figure (3.2) showing the effects of reservoir water treated by activated carbon (0.250 mm) on the oil recovery for sandstone core samples in relation to time and temperature.

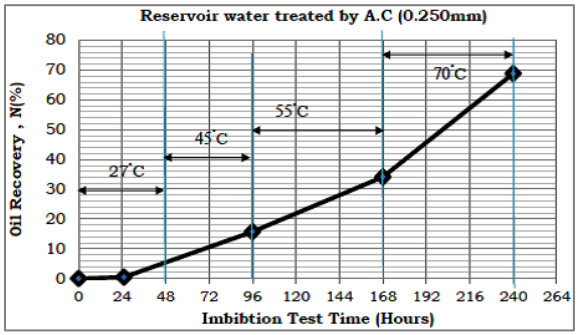

Figure (3.2) Effects of Reservoir Water Treated with Activated Carbon (0.250 mm) on Oil Recovery of Sandstone Core Samples Related with Time and Temperature

(ii) Second Scenario; Reservoir Water was Treated by Activated Carbon (0.125 mm)

The spontaneous imbibition test was done by the reservoir water treated by activated carbon with size of 0.125 mm, and the used sample was (C-003). At the beginning the temperature was about (27 ˚C) for a period time of 24 hours, thus, the oil extraction rate (OER) was (0.40 %), by increasing the temperature to (45˚C) for a period of 72 hours, (OER) was increased to (15.73 %). Then the test was carried out for more addition 72 hours at the same temperature, in this case (OER) was (34.13 %). Finally. the temperature was raised to (70 ˚C) for 72 hours and (OER) was (68.87 %). Figure (3.3) show the effects of reservoir water (untreated) on the oil recovery for sandstone core samples as function to time and temperature.

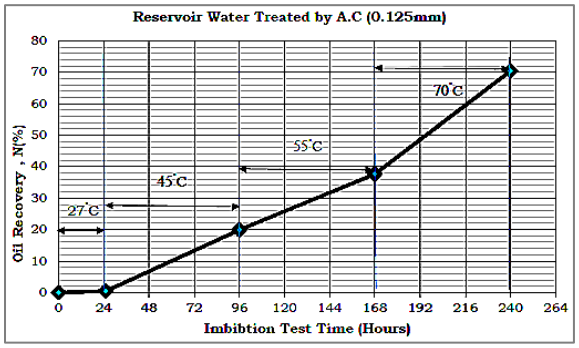

Figure (3.3) Effects of Reservoir Water Treated with Activated Carbon (0.125 mm) on Oil Recovery of Sandstone Core Samples Related with Time and Temperature

(iii) Third Scenario; Reservoir Water was Treated by Activated Carbon (0.063 mm)

The spontaneous imbibition test was carried out by the reservoir water treated by activated carbon with size of 0.063 mm, and the used sample was (C-004), starting at of temperature was about (27 ˚C) for a period time of 24 hours, thus, the oil extraction rate

(OER) was (0.32 %). Increasing the temperature to (45 ˚C) for a period of 72 hours, (OER) resulted in an increase of (10.01 %), then the test was carried out for addition 72 hours at the same temperature, in this case (OER) was (24.01 %). Finally. the temperature was raised to (70˚C) for 72 hours and (OER) was (53.10 %). Figure (3.4) show the effects of reservoir water (untreated) on the oil recovery for sandstone core samples in relation to time and temperature.

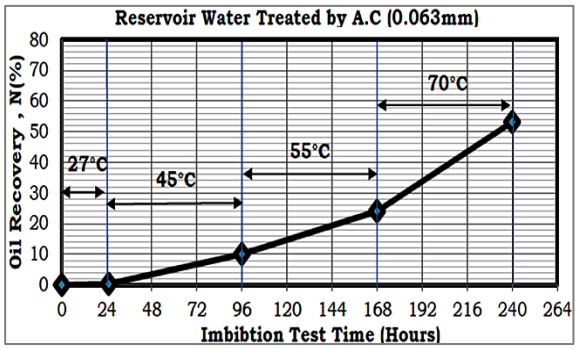

Figure (3.4) Effects of Reservoir Water Treated with Activated Carbon (0.063 mm) on Oil Recovery of Sandstone Core Samples Related with Time and Temperature

3.4.3 Comparison between the Effects of Oil Recoveries (Sandstone Rocks)

The spontaneous imbibition test of sandstone samples was performed by untreated reservoir water and treated reservoir water at different volumes of activated carbon, using different temperatures and different interval times. Figure (3.5)` show the comparison in terms of oil extraction.

The extraction of oil in spontaneous imbibition test using the sample (C-001) which was carried out using a natural reservoir water (untreated reservoir water) with low rates despite the slight, was increased gradually when a different temperatures were applied . The maximum oil extraction reached (46.8 %) at the maximum temperature of (70 °C) . Using the smaller size of 0.063 mm of activated carbon on the sample (C-004) gave the least effect for the extraction process when a treated water was used . Moreover, the reached maximum value of extraction (53.1 %) at maximum temperature. This difference is small compared to the effect of the natural reservoir water. The rate of oil extraction became higher by using a larger sizes of activated carbon. When the samples (C-002 and C-003) were used, the case was different. The oil extraction was increased at higher rates compared to the other two samples at two temperatures (55°C and 70 °C). They had a very close maximum extraction (70.41 % and68.87 %). It turns out that the treated water has higher efficiency despite the difference

in its efficacy according to the volume. Activated carbon played a major role in

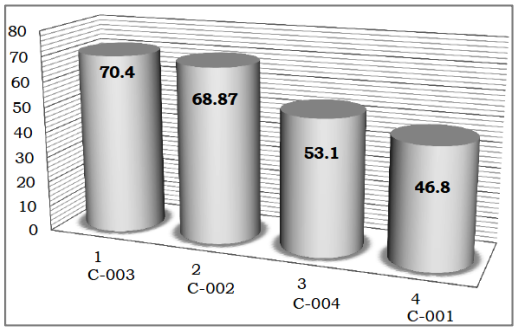

Figure (3.5) Comparison Between Oil Recoveries for Sandstone Cores

changing the properties of reservoir water and changing amount of some other elements. Also the terrible change in the smell of the water can be noticed. After the treatment process, the smell of sulfur became almost non-existent, compared to that of untreated reservoir water.

4. Conclusions and Future Recommendation

4.1 Conclusions

(i) The oil recovery rates are highly related to the applied temperature, meaning that the oil recovery rates is directly proportional to the temperature; (ii) Activated carbon with a volume of (0.125 mm) has the highest efficiency as it gave the highest rate of oil recovery compared to volume (0.250 mm). While volume of (0.063 mm) had the lowest oil recovery rate; (iii) The overall conclusion is that the reservoir treated water with activated carbon is slightly better compared to the natural reservoir water (untreated reservoir water) in terms of oil recovery.

4.2 Future Recommendation

(i) Another type of reservoir rocks can be used, such as comparing the carbonate rocks to the sandstone rocks; (ii) Different sizes of activated carbon can be used in order to compare the obtained results to the results in the present study; (iii) A more quantities of activated carbon in the treated water can be used; (iv) The properties of activated carbon should be carefully studied; (v) The reservoir water treatment by the spontaneous imbibition test may be considered at a different test time compared to those used in the present study; (vi) A known combination and properties of a different kind of crude oil can be used in order to compare the obtained data with the results of the crude oil kind used in the present study.

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